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THE SYNTHESIS AND CHARACTERIZATION OF 1,1-(P(C6H5)3)2-1-H-1,2,4—ETC(U)
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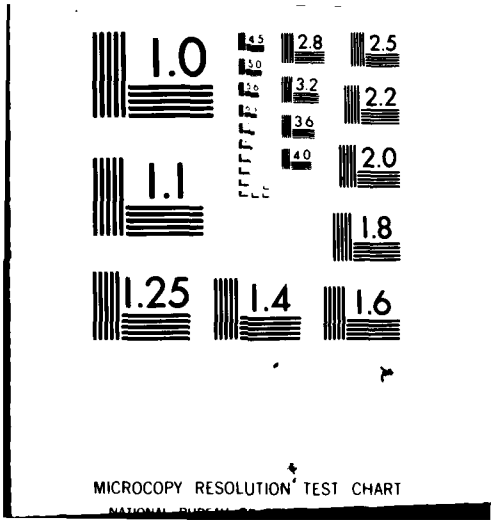
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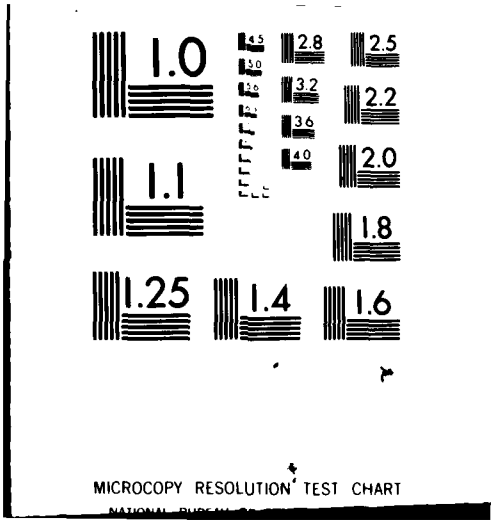
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TECHNICAL REPORT NO. 113

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By

John D. Hewes, Carolyn B. Knobler, M. Frederick Hawthorne*

14 Feb. 81

Prepared for Publication

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Department of Chemistry
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Los Angeles, California 90024

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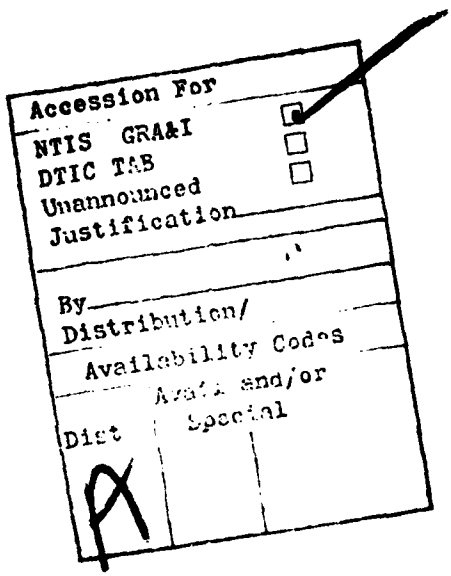

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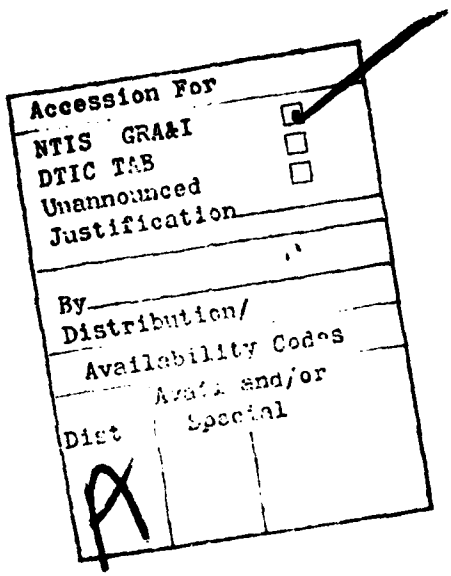

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Icosahedral hydridophosphinorhodacarboranes of general formula $C_2B_9H_{11}RhH(P(C_6H_5)_3)_2$ have been shown to catalytically hydrogenate blocked olefins[1a,b] and an intensive study has focused on the elucidation of catalytic pathways[2] and the associated structural chemistry[3] unique to catalytically active metallocarboranes. A successful effort to synthesize 10-[4] and 11-[5] vertex catalyst precursors through routes analogous to the 12-vertex synthesis, i.e., by reaction of the monoanions $C_2B_7H_{12}^-$ and $C_2B_8H_{11}^-$ with $[(P)C_6H_5)_3)_3RhCl]$, prompted an examination of synthetic routes

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The thermally unstable $[(CH_3)_4N]^+[C_2B_{10}H_{13}]^-$ rearranges at 100°C to the stable $[(CH_3)_4N]^+[9,12\text{-}C_2B_{10}H_{13}]^-$. The structural differences between the two isomers probably exist in the position of the carbon atoms, and in the tentative assignment of a B-H-B bridge in the unstable isomer[10]. In addition, the reactivity of the isomers toward metal complex formation was found to differ significantly.

Refluxing methanolic solutions of the stable $[(CH_3)_4N]^+$ $[9,12\text{-}C_2B_{10}H_{13}]^-$ with $[(P(C_6H_5)_3)_3\ RhCl]$ produced no metallocarboranes, as deduced from the lack of a ca. 2500 $cm.^{-1}$ B-H stretching band in the I.R. spectra of the observed products.

Reaction of the unstable $[(CH_3)_4N]^+[C_2B_{10}H_{13}]^-$ with $[(P(C_6H_5)_3)_3RhCl]$[11] produced closo-1,1-$(P(C_6H_5)_3)_2$-1-H-1,2,4-$RhC_2B_{10}H_{12}$ (I), which was found to catalytically hydrogenate blocked olefins under mild conditions, and which also exhibited a structural chemistry different from previously reported[12-14] 13-vertex metallocarboranes derived from the $C_2B_{10}H_{12}^{2-}$ ion. I was characterized by $^{31}P\{^1H\}$, ^1H, and ^{11}B NMR prior to the x-ray crystal structure analysis.

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For the x-ray crystal structure analysis, $\underline{\underline{I}}$ was prepared as described and purified by recrystallizations from THF/n-heptane and THF/ethanol. Suitable crystals of monoclinic, $P2_1/n$ symmetry were grown from 1,2-dichloroethane/n-pentane by vapor diffusion. As shown in Figure 1, the cage geometry of $\underline{\underline{I}}$ differs from the structure

Figure 1

of 1-(π-cyclopentadienyl)-1,2,4-$CoC_2B_{10}H_{12}$ reported by Churchill[14]. The most obvious feature is the lack of a bonding interaction between B(3) and B(8) with an internuclear distance of 2.166 Å; this compares with a value of 2.082 Å, which Churchill emphasized was abnormally long, and represents a significant departure from the normally triangulated polyhedral carborane systems. The B(9)···C(2) diagonal in the C(2)-B(3)-B(9)-B(8) system is 2.720 Å, in the C(2)-B(7)-B(12)-B(8) system the diagonal C(2)···B(12) is 2.841 Å, and in the B(10)-B(9)-B(3)-C(4) system the B(10)···B(3) diagonal is 2.938 Å.

The four boron atoms in the top belt of 1-(π-cyclopentadienyl)-1,2,4-$CoC_2B_{10}H_{12}$ were observed to be exactly coplanar, with C(2) above and C(4) below that plane. The structure of $\underline{\underline{I}}$ varies in this respect, with small deviations from planarity observed in the top belt, but with the locations of C(2) and C(4) exhibiting greater least squares deviations from planarity than B(3), B(5), B(6), and B(7). The entire top belt is distorted to accommodate the perturbed 13-vertex system.

The structural chemistry of $\underline{\underline{I}}$ is unique when compared to the systems previously reported [12-14]. The variable temperature ^{11}B NMR indicated no cage fluxionality, and no structural changes were observed for samples which had been refluxed in THF for 3 hrs. In fact, thermal isomerization was not observed when $\underline{\underline{I}}$ was heated *in vacuo* as a solid to its decomposition temperature (490K) at 25° increments and followed by IR spectroscopy.

Variable temperature ^{1}H and $^{31}P\{^{1}H\}$ NMR spectra of $\underline{\underline{I}}$ indicated hindered rotation of the metal vertex about the planar hexagonal bonding face of the carborane cage; this is consistent with previous conclusions based on studies of icosahedral *closo*-bisphosphinometallocarboranes[15].

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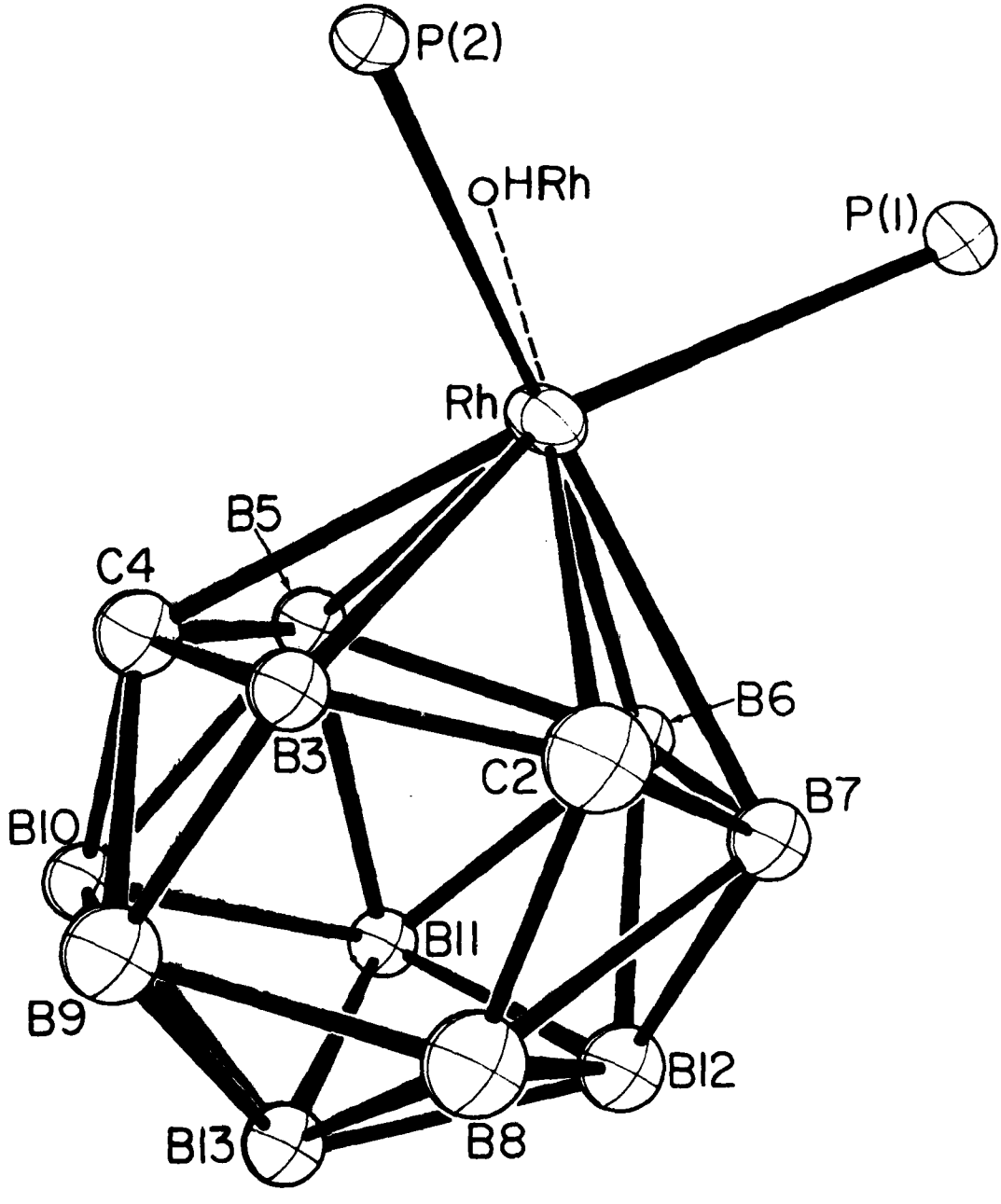
ACKNOWLEDGEMENTS

Figure 1. The molecular structure of 1,1-$(P(C_6H_5)_3)_2$-1-H-1,2,4-$RhC_2B_{10}H_{12}$•1.5 $C_2H_4Cl_2$. The molecules of solvation, the phenyl rings, and the terminal boron hydrides have been omitted for clarity. The metal hydride was located on the difference maps only.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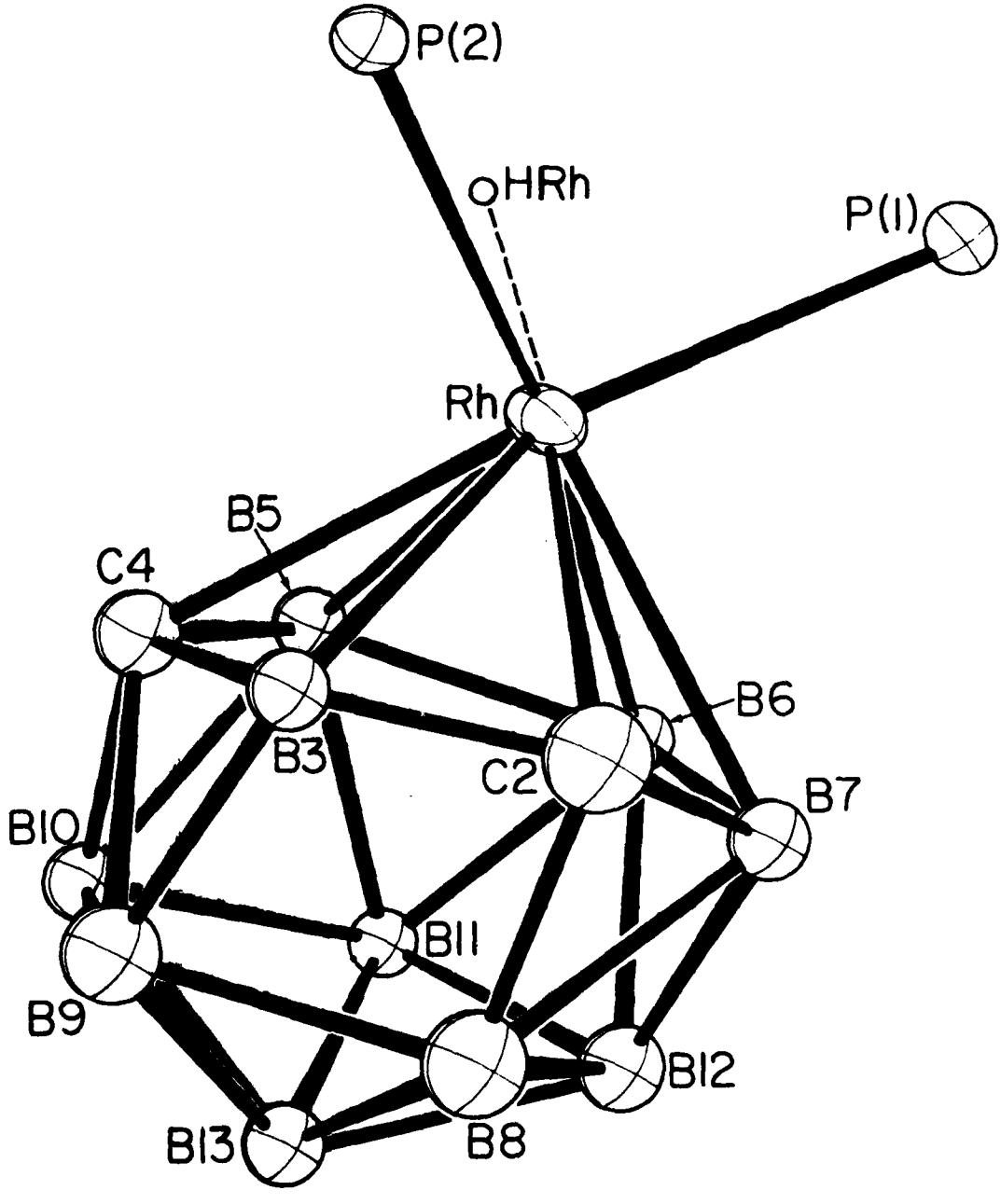
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